

X-Ray Fluorescence Spectrometry - XRF

Introduction

X-ray secondary-emission spectrometry or X-Ray Fluorescence spectrometry (XRF) is a nondestructive method of qualitative and quantitative analysis for elemental composition of samples. XRF analysis is based on the measurement of wavelengths and intensities of x-ray spectral lines emitted by secondary excitation.

Applied Research Laboratories 8420 X-ray fluorescence spectrometer was installed in the X-ray Laboratory on the *JOIDES Resolution (JR)* prior to the Leg 102 transit to Portugal. During the Ocean Drilling Program (ODP), scientists sailing on the *JR* analyzed almost 4000 samples from 56 legs before the spectrometer was removed from the ship after Leg 189.

XRF Data Acquisition

The ARL 8420 was a fully automated, wavelength-dispersive spectrometer using a 3-kW rhodium x-ray tube as the excitation for both major oxides and trace elements. Scientists and technicians on Legs 109 and 111 developed the first set of ODP XRF standard operating procedures. Few changes were made to the XRF hardware or the standard operation procedures during the period that the XRF system was on the *JR*. A summary of XRF data operations during the ODP is found below.

Table 1. Summary of XRF operations.

XRF Hardware	Legs
ARL 8420, 1 goniometer, PDP-11 computer	106, 109, 111, 113, 114, 115, 116, 118, 119
ARL 8420, 2 goniometers, PDP-11 computer	120, 121, 123, 124, 125, 126, 127, 128, 130, 132, 134, 135, 136, 137, 138, 139, 140, 141, 142, 143, 144, 145, 147, 148
ARL 8420, 2 goniometers, PC computer, automated data reduction	149, 150, 151, 152, 153, 155, 157, 158, 161, 163, 164, 166, 168, 169, 171, 173, 176, 178, 179, 180,
ARL 8420, 2 goniometers, PC Pentium computer	183, 185, 187

In Table 2 below, the major oxides and trace elements routinely collected with the XRF are listed with an example of the operating parameters of the system (Leg 125). Parameters such as Line, Crystal, and Detector did not change except when the Krypton detector was replaced by an FPC on Leg 136. The Collimator setup should not have been changed much; however, some legs designated the settings as fine and coarse; others designated the settings as fine and medium. Peak Angle and Background Offset would change slightly with each calibration. Background angles were adjusted to avoid overlap from other elements.

Table 2. ARL 8420 XRF spectrometer operating parameters.

Oxide or element	Line	Crystal	Detector ^a	Collimator	Peak angle (degrees)	Background offset (degrees)	Count time on peak (s)	Count time on background (s)
SiO ₂	K α	PET(002)	FPC	Coarse	109.25	0	40	0
TiO ₂	K α	LiF(200)	FPC	Fine	86.14	0	40	0
Al ₂ O ₃	K α	PET(002)	FPC	Coarse	145.27	0	100	0
Fe ₂ O ₃	K α	LiF(200)	FPC	Fine	57.52	0	40	0
MnO	K α	LiF(200)	KrSC	Fine	62.98	0	40	0
MgO	K α	TLAP	FPC	Coarse	44.87	± 0.80	200	200
CaO	K α	LiF(200)	FPC	Coarse	113.16	0	40	0
Na ₂ O	K α	TLAP	FPC	Coarse	54.71	-1.20	200	200
K ₂ O	K α	LiF(200)	FPC	Fine	136.65	0	40	0
P ₂ O ₅	K α	GE(111)	FPC	Coarse	140.94	0	100	0
Rh	K Compton	LiF(200)	Scint	Fine	18.59	0	100	0
Nb	K α	LiF(200)	Scint	Fine	21.37	± 0.35	200	200
Zr	K α	LiF(200)	Scint	Fine	22.53	± 0.35	100	100
Y	K α	LiF(200)	Scint	Fine	23.78	± 0.40	100	100
Sr	K α	LiF(200)	Scint	Fine	25.13	± 0.40	100	100
Rb	K α	LiF(200)	Scint	Fine	26.60	± 0.60	100	100
Zn	K α	LiF(200)	Scint	Coarse	41.79	± 0.40	60	60
Cu	K α	LiF(200)	Scint	Fine	45.02	± 0.40	60	60
Ni	K α	LiF(200)	Scint	Coarse	48.67	± 0.60	60	60
Cr	K α	LiF(200)	FPC	Fine	69.35	± 0.50	60	60
Fe	K α	LiF(220)	FPC	Fine	85.37	-0.40 +0.70	40	40
V	K α	LiF(220)	FPC	Fine	122.84	-0.50	60	5060
TiO ₂	K α	LiF(200)	FPC	Fine	86.14	± 0.50	40	40
Ce	L α	LiF(220)	FPC	Coarse	127.92	± 1.50	100	100
Ba	L β	LiF(220)	FPC	Coarse	128.53	± 1.50	100	100

Notes: All elements measured using a rhodium X-ray tube operated at 60 kV and 50 mA.

^a FPC = flow proportional counter using P₁₀ gas; KrSC = sealed krypton gas counter; Scint = NaI scintillation counter.

Sample preparation

Most samples analyzed by the ODP XRF were first powdered because of the inhomogeneity of rock and sediment samples. The powder was processed into fused glass disks to be run for major oxide analyses, and pressed pellets to be run for trace element analyses. The descriptions of sample preparation varied some between legs; the descriptions below are representative of the general procedures.

Hard Rock samples, approximately 10 cm³ of rock, had saw marks and unwanted material removed by wet-grinding on a silicon carbide disk mill. The samples were ultrasonically washed in distilled water then methanol for 10 minutes and dried at 110 °C for at least two hours. Larger pieces were reduced to less than 1 cm diameter by crushing between two plastic disks in a hydraulic press. Powders were produced by grinding pieces less than 1 cm in diameter in a Spex Shatterbox, using a tungsten carbide grinding vessel, for 60-120 s, depending on the size of the vessel. The powder was transferred to clean paper, and then to a sample vial and labeled. The vessel was thoroughly cleaned and prepared for another sample.

Lithified sediment samples were treated like hard rock samples except that the oven drying was replaced by freeze-drying the sample for at least 12 hours in the freeze drier. After the sample was dried, it was ground and treated in the same manner as the hard rock sample.

Unlithified (mud) samples were more problematic. Sometimes the mud was washed to remove chloride, and then freeze-dried. Other times the mud was just freeze-dried because it was believed that washing would remove other elements, not just the chloride contamination. After the sample was dried, it was ground and treated in the same manner as the hard rock sample.

Approximately 1.5 g of the rock powder was carefully weighed and ignited in an ash furnace for at least 5 hours at 1000 °C for hard rock and 900 °C for sediments. If the sample likely contained muscovite, biotite, amphibole or carbonates, the sample was ignited for at least 6 hours. Because the powders were dried before ignition, the loss values due to the amount of adsorbed water (H_2O^-) were assumed to be negligible.

Fused glass disks were created for major oxide analysis in order to reduce matrix effects and variations in background (Claisse, 1956; Rose et al., 1962; Norrish and Hutton, 1969). These disks were made by mixing 7.20 g (20% La_2O_3) lithium tetraborate flux with 0.600 g of ignited rock powder. This sample/flux mixture was melted at 1030 °C in platinum-gold crucibles for 6-10 minutes and poured into Pt-Au molds using a modified Claisse Fluxer apparatus. The 12:1 flux-to-sample ratio had been found to sufficiently reduce matrix effects to the point where matrix corrections were unnecessary for normal basaltic to granitic composition ranges.

Trace Elements

Pressed pellets were used for the trace element analyses. Pressed-powder pellets were made by mixing 7 g of fresh rock powder with 30 drops of polyvinyl alcohol binder, then pressing the mixture into an aluminum cap with 7 tons of pressure. A minimum of 5 g of sample usually guaranteed the pellet would be 'infinitely thick' for rhodium K-series radiation.

To compute trace element concentrations from measured X-ray intensities, an off-line calculation program was written J.W. Sparks based on routines modified from Norrish and Chappell (1967) and Reynolds (1967). After the computer and software upgrade on Leg 149, trace element calculations were done by the data acquisition software.

Calibration

Most XRF analytical methods are based on comparison of the unknown sample's line intensities to one or more well-characterized standards. The standards must be similar to the samples in physical form, elemental concentrations, and matrix composition. The calibration for a leg was performed using standards chosen to best provide the range of values that were expected from the cored rock. Table 3 lists the standards most often used on the *JR*. These standards were chosen because of their similarity to oceanic basalts, granites and ultramafic rocks.

Oceanic sediments are harder to characterize because of the variety of materials that make up the sedimentary column, e.g., organic material, calcium carbonate, clay, or silica. Developing standards that would span the expected range of values was very difficult.

Table 3. Standard Reference Materials

XRF Standard Name	Replicate	Rock Type	Comment
152-11	A	MORB	
152-75	A	MORB	
AGV-1	A	Andesite	
AII-92-29-1	A	MORB/Basalt	
AII-92-29-1	X	MORB/Basalt	
AMERSIL	A	Blank for Bkg.	
BA-0500	A	Line Overlap Std.	
BA-1000	A	Line Overlap Std.	
BA-2000	A	Line Overlap Std.	
BAS-140	A	Diabase (504B)	
BAS-148	A	Basalt	
BE-N	A	Basalt	
BE-N (BR)	A	Alkali Basalt	

XRF Standard Name	Replicate	Rock Type	Comment
BE-N (PP)	A	Basalt	Pressed pellet
BHVO-1	A	Tholeiite/Basalt	
BHVO-1	B		
BHVO-1	TR1	Pressed pellet	
BHVO-2	A	Basalt	
BIR-1	A	Basalt	
BOB-1	A	MORB	
BR-1	A	Basalt	
CaCO3	A	Blank for Bkg.	ULTREX
CE-0500	A	Line Overlap Std.	
CE-1000	A	Line Overlap Std.	
CE-2000	A	Line Overlap Std.	
DNC-1	A	Diabase	
DR-N	A	Diorite	Leg 173, 12:1 ratio with Flux VII, NT-2100 bead
FE2O3	A	Blank for Bkg.	
Flux IX	A	Blank bead	
G-2	A	Granite	
G-2 (PP)	A	Granite	Pressed pellet
GBM-1	A	Garnet	
JA-1	A	Andesite	
JA-2	A	Andesite	
JA-3	A	Andesite	
JB-1A	A	Basalt	
JB-2	A	Basalt	
JB-3	B	Basalt	
JF-1	A	Feldspar	
JF-2	A	Feldspar	
JG-1a	A	Granite	MAJOR
JG-1a	B	Granodiorite	TRACE
JG-2	A	Granite	
JG-3	A	Granodiorite	
JGB-1	A	Gabbro	
JP-1	A	Peridotite	
JR-1	A	Rhyolite	
JR-2	A	Rhyolite	
K1919	A	Tholeiite	
L12B407	A	Blank for Bkg.	
MAG-1	A	Sediment	
MGO	A	Blank for Bkg.	
Mica-Fe	A	Biotite	
Mica-Mg	A	Phlogopite	
MRG-1	A	Gabbro	
NBS-1c	A	Limestone	
NBS-278	A	Obsidian	
NBS-688	A	Basalt	
NIM-D	A	Dunite	
NIM-P	A	Pyroxenite	
PCC-1	A	Peridotite	
RB-0500	A	Line Overlap Std.	
RB-1000	A	Line Overlap Std.	
RB-2000	A	Line Overlap Std.	
RGM-1	A	Rhyolite	
SCo-1	A	Shale	
SCo-1	B	Cody Shale	Pressed Pellet
SCo-1	TR1	Pressed pellet	
SCo-1	X	Cody Shale	
SDC-1	A	Mica Schist	
SR-0500	A	Line Overlap Std.	
SR-1000	A	Line Overlap Std.	
SR-2000	A	Line Overlap Std.	
STM-1	A	Syenite	
SY-2	A	Syenite	
TI-0500	A	Line Overlap Std.	
TI-1000	A	Line Overlap Std.	
TIO2-9.3%	A	Line Overlap Std.	
UB-N	A	Serpentinite	Leg 173, 12:1 ratio with Flux VII, NT-2100 bead
UB-N (PP)	A	Serpentinite	Pressed pellet
V-0500	A	Line Overlap Std.	
V-1000	A	Line Overlap Std.	
V-2000	A	Line Overlap Std.	
W-2	A	Diabase	
Y-0500	A	Line Overlap Std.	
Y-1000	A	Line Overlap Std.	
Y-2000	A	Line Overlap Std.	

Data Archive

Pre-JANUS Archive

Major oxide and trace element data from XRF analyses were logged on logsheets. The logsheets were brought back to ODP/TAMU at the end of each leg, entered into an S1032 database, and microfilmed for archival purposes. The S1032 database was used to store data until Leg 134. Starting with Leg 134, the data were saved in files which were brought back at the end of each leg and archived on the ODP/TAMU servers.

Migration of XRF to JANUS

The data model for XRF data can be found in Appendix I. Included are the relational diagram and the list of the tables that contain data pertinent to XRF, the column names and the definition of each column attribute. ODP Information Services Database Group was responsible for the migration of pre-Leg 171 data to Janus. An additional column of information was added to the XRF_SAMPLE table after the migration of data. This column, Sample_type, was added to allow scientists to describe the type of rock that was analyzed. In turn, scientists would be able to extract data based on specific rock types. The sediment or rock designations were not usually stored in the original data files, and it was necessary to extract that information from the Initial Report volumes. Due to constraints of time, it was not possible to complete this part of the migration.

Janus XRF Data Format

XRF major oxide and trace element data can be retrieved from Janus Web using a predefined query. The X-Ray Fluorescence (XRF) query webpage allows the user to extract data using the following variables to restrict the amount of data retrieved: leg, site, hole, core, section, depth range or latitude and longitude range. Often, replicate samples were analyzed for the major oxides; those data, when available, were uploaded separately. The web query reports the replicate data on separate lines. In addition, the trace element data will also be reported on a separate line.

Table 4 lists the data fields retrieved from the Janus database for the XRF predefined query. The first column contains the data item; the second column indicates the Janus table or tables in which the data were stored; the third column is the Janus column name or the calculation used to produce the value. Appendix II contains additional information about the fields retrieved using the Janus Web XRF query and the data format for the archived ASCII files.

Table 4. X-Ray Fluorescence (XRF) query

Item Name	Janus Table	Janus Column Name and Calculation
Leg	SECTION	Leg
Site	SECTION	Site
Hole	SECTION	Hole
Core	SECTION	Core
Type	SECTION	Core_type
Section	SECTION	Section_number
Top (cm)	SAMPLE	Top_interval x 100
Bottom (cm)	SAMPLE	Bottom_interval X 100
Depth (mbsf)	DEPTH_MAP, SAMPLE	DEPTH_MAP.Map_Interval_Top + SAMPLE.Top_interval
Run	XRF_SAMPLE	XRF_Run_Identifier
Replicate	XRF_SAMPLE	XRF_Replicate
Bead Loss on Ignition	XRF_SAMPLE	Bead_LOI
Silica – SiO ₂ (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code – SiO ₂ ::XRF_Analysis_Result
Titanium Oxide – TiO ₂ (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code – TiO ₂ ::XRF_Analysis_Result
Aluminum Oxide – Al ₂ O ₃ (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code – Al ₂ O ₃ ::XRF_Analysis_Result

Item Name	Janus Table	Janus Column Name and Calculation
Iron Oxide - Fe ₂ O ₃ * (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Fe2O3::XRF_Analysis_Result
Manganous Oxide - MnO (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - MnO::XRF_Analysis_Result
Magnesium Oxide - MgO (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - MgO::XRF_Analysis_Result
Calcium Oxide - CaO (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - CaO::XRF_Analysis_Result
Sodium Oxide - Na ₂ O (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Na2O::XRF_Analysis_Result
Potassium Oxide - K ₂ O (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - K2O::XRF_Analysis_Result
Phosphorus Pentoxide - P ₂ O ₅ (wt %)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - P2O5::XRF_Analysis_Result
Niobium - Nb (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Nb::XRF_Analysis_Result
Zirconium - Zr (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Zr::XRF_Analysis_Result
Yttrium - Y (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Y::XRF_Analysis_Result
Sulfur - S (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - S::XRF_Analysis_Result
Strontium - Sr (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Sr::XRF_Analysis_Result
Rubidium - Rb (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Rb::XRF_Analysis_Result
Scandium - Sc (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Sc::XRF_Analysis_Result
Molybdenum - Mo (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Mo::XRF_Analysis_Result
Beryllium - Be (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Be::XRF_Analysis_Result
Thorium - Th (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Th::XRF_Analysis_Result
Cobalt - Co (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Co::XRF_Analysis_Result
Gadolinium - Gd (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Gd::XRF_Analysis_Result
Dysprosium - Dy (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Dy::XRF_Analysis_Result
Erbium - Er (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Er::XRF_Analysis_Result
Ytterbium - Yb (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Yb::XRF_Analysis_Result
Hafnium - Hf (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Hf::XRF_Analysis_Result
Lead - Pb (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Pb::XRF_Analysis_Result
Gallium - Ga (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Ga::XRF_Analysis_Result
Zinc - Zn (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Zn::XRF_Analysis_Result
Copper - Cu (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Cu::XRF_Analysis_Result
Nickel - Ni (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Ni::XRF_Analysis_Result
Chromium - Cr (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Cr::XRF_Analysis_Result
Vanadium - V (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - V::XRF_Analysis_Result
Cerium - Ce (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Ce::XRF_Analysis_Result
Barium - Ba (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Ba::XRF_Analysis_Result
Cesium - Cs (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Cs::XRF_Analysis_Result
Lanthanum - La (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - La::XRF_Analysis_Result
Neodymium - Nd (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Nd::XRF_Analysis_Result
Samarium - Sm (ppm)	XRF_SAMPLE_ANALYSIS	XRF_Analysis_Code - Sm::XRF_Analysis_Result
Sample Type	XRF_SAMPLE_TYPE	Sample_Type
Comment	XRF_SAMPLE	XRF_Comment

Data Quality

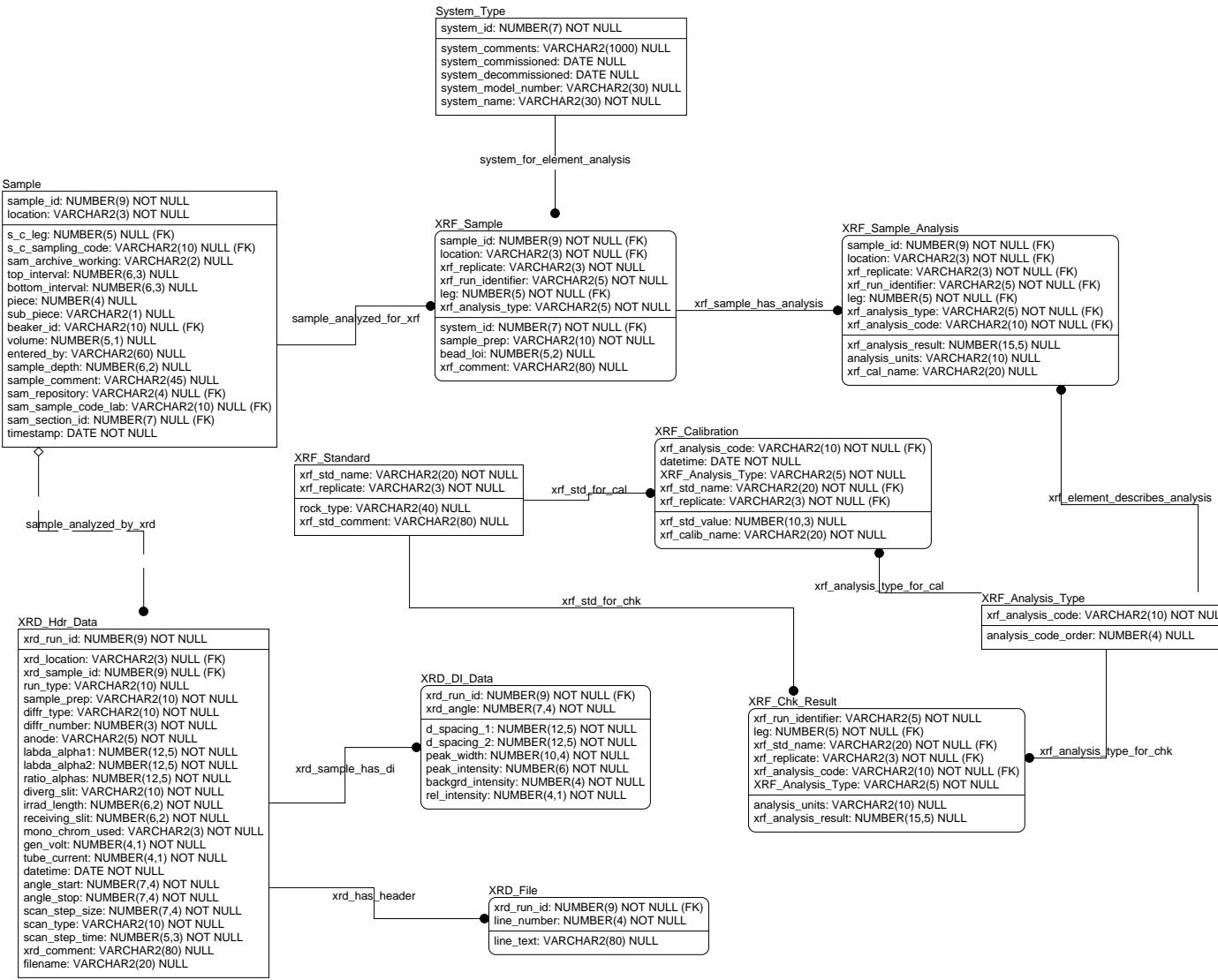
The XRF data were very high quality, even though the ship environment made getting accurate measurements more difficult. XRF analytical methods are based on comparison of the unknown sample's line intensities to one or more well-characterized standards. For this reason, XRF analyses done on volcanic, mafic and ultramafic samples are the highest quality because there are standards that characterize the range of elemental concentrations most often found in these types of rocks. It is more difficult to create standards that would encompass the range of elemental concentrations found in sedimentary environments. Migration of the sample type data was completed for 20 of the 56 legs.

References

Bertin, E.P., 1975, Principles and Practice of X-Ray Spectrometric Analysis, Second Edition, Plenum Press, New York.

Claisse, F., 1956, Accurate X-ray fluorescence analysis without internal standard. *Quebec Dept. Mines Press Release 327.*

- Norrish, K. and Chappell, B.W., 1967. X-ray fluorescence spectrometry. In Zussman, J., (Ed), *Physical methods in Determinative Mineralogy*: New York (Acad. Press), 161-214.
- Norrish, K. and Hutton, J.T., 1969. An accurate X-ray spectrographic method for the analysis of a wide range of geological samples. *Geochim. Cosmo. Acta*, 33:431-453.
- Rose, J.J., Adler, I., and Flanagan, F.J., 1962. Use of La_2O_3 as a heavy absorber in X-ray fluorescence analysis of silicate rocks. U.S. Geol. Surv. Prof. Paper, 450B:80-82.
- Shipboard Scientific Party, 1988. Introduction and Explanatory Notes. In Becker, K., Sakai, H., et al., *Proc. ODP, Init. Repts. (Pt. A)*, 111:9-10, College Station, TX (Ocean Drilling Program).
- Thompson, G. and Bankston, C.C., 1970. Sample contamination from grinding and sieving determined by emission spectroscopy. *Appl. Spectros.*, 24:210-219.



Appendix I: Janus Data Model – X-Ray Fluorescence - XRF

X-Ray Fluorescence - XRF		
Table Name	Column Name	Column Comment
XRF_Sample	sample_id	Oracle-generated sequence number that with <i>location</i> uniquely identifies a sample.
	location	Code that indicates which Janus application assigned the sample_id. Values are SHI (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository, WCR (West Coast Repository) and BCR (Bremen Core Repository). Used with <i>sample_id</i> to uniquely identify a sample.
	XRF_replicate	Identifier for each replicate of a sample to allow all to be entered into the database.
	XRF_run_identifier	Operator-assigned run identifier. Must be unique during a leg.
	leg	Number identifying the cruise for which data were entered into the database.
	XRF_analysis_type	Type of analysis performed on an XRF sample -- MAJOR oxide, TRACE element.
	system_id	Unique identifier for a system of equipment used to collect data.
	sample_prep	Type of preparation used for a sample - a fused glass disc (bead) or pressed pellet (pellet).
	bead_loi	Loss on Ignition. The percentage of weight lost after igniting the XRF bead [(post_ign_sample_wt/pre_ign_sample_wt)-1]*(-100).
	XRF_comment	General comment about sample or analysis.
XRF_Sample_Analysis	sample_id	Oracle-generated sequence number that with <i>location</i> uniquely identifies a sample.
	location	Code that indicates which Janus application assigned the sample_id. Values are SHI (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository, WCR (West Coast Repository) and BCR (Bremen Core Repository). Used with <i>sample_id</i> to uniquely identify a sample.
	XRF_replicate	Identifier for each replicate of a sample to allow all to be entered into the database.
	XRF_run_identifier	Operator-assigned run identifier. Must be unique during a leg.
	leg	Number identifying the cruise for which data were entered into the database.
	XRF_analysis_type	Type of analysis performed on an XRF sample -- MAJOR oxide, TRACE element.
	XRF_analysis_code	The code for the element or oxide being analyzed.
	XRF_analysis_result	Analytical result for an analysis code.
	analysis_units	Measurement units used for an analysis. Major oxides - wt%; Trace elements - ppm.
	XRF_cal_name	The same description as the attribute XRF_calib_name, but allowed to be null.
XRF_Analysis_Type	XRF_analysis_code	The code for the element or oxide being analyzed.
	analysis_code_order	Used to determine the order that analysis codes will appear on a spreadsheet or report.
XRF_Sample_Type	sample_type_id	ID assigned to the rock type.
	sample_type	Rock type name, e.g., Basalt, Granite, Oxide Gabbro.
XRF_Standard	XRF_std_name	The name of an XRF standard.
	XRF_replicate	Identifier for each replicate of a sample to allow all to be entered into the database.
	rock_type	Description of the rock type or material of the standard.
	XRF_std_comment	Comment about an XRF standard.
XRF_Calibration	XRF_analysis_code	The code for the element or oxide being analyzed
	datetime	Generic date/time. Often used for keys when multiple comments, etc can be entered.
	XRF_Analysis_Type	Type of analysis performed on an XRF sample -- MAJOR oxide, TRACE element.
	XRF_std_name	The name of an XRF standard
	XRF_replicate	Identifier for each replicate of a sample to allow all to be entered into the database.
	XRF_std_value	The expected results for a element in an XRF standard
	XRF_calib_name	Name associated with a particular calibration, since the XRF may run multiple calibrations at any time.
XRF_Chk_Result	XRF_run_identifier	Operator-assigned run identifier. Must be unique during a leg.
	leg	Number identifying the cruise for which data were entered into the database.
	XRF_std_name	The name of an XRF standard.
	XRF_replicate	Identifier for analysis replicates of a standard to allow all to be entered into the database.
	XRF_analysis_code	The code for the element or oxide being analyzed.
	XRF_Analysis_Type	Type of analysis performed on an XRF sample -- MAJOR oxide, TRACE element.
	analysis_units	Measurement units used for an analysis. Major oxides - wt%; Trace elements - ppm.
	XRF_analysis_result	Analytical result for an analysis code.

X-Ray Fluorescence - XRF

Table Name	Column Name	Column Comment
Section	section_id	Unique Oracle-generated sequence number to identify each section. This is done because of the physical subsection / zero section problems. In adding new sections, deleting sections or changing sections - don't want to have to renumber.
	leg	Number identifying the cruise for which data were entered into the database.
	site	Number identifying the site from which the core was retrieved. A site is the position of a beacon around which holes are drilled.
	hole	Letter identifying the hole at a site from which a core was retrieved or data were collected.
	core	Sequential numbers identifying the cores retrieved from a particular hole. Cores are generally 9.5 meters in length, and are numbered serially from the top of the hole downward.
	core_type	A letter code identifying the drill bit/coring method used to retrieve the core.
	section_number	Cores are cut into 1.5 m sections. Sections are numbered serially, with Section 1 at the top of the core.
	section_type	Used to differentiate sections of core (S) from core catchers (C). Previously core catchers were stored as section CC, but in Janus core catchers are given the next sequential number from the last section recovered.
	curated_length	The length of the section core material, in meters. This may be different than the liner length for the same section. Hard rock cores will often have spacers added to prevent rock pieces from damaging each other.
	liner_length	The original length of core material in the section, in meters. Sum of liner lengths of all the sections of a core equals core recovery.
	core_catcher_stored_in	Sometimes the core catcher is stored in a D tube with a section. core_catcher_stored_in contains the section number of the D tube that holds the core catcher.
	section_comments	Comments about this section
Sample	sample_id	Oracle-generated sequence number that with location uniquely identifies a sample.
	location	Code that indicates which Janus application assigned the sample_id. Values are SHI (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository, WCR (West Coast Repository) and BCR (Bremen Core Repository). Used with sample_id to uniquely identify a sample.
	s_c_leg	Number identifying the cruise for which data were entered into the database. Foreign key used with s_c_sampling_code to link samples with a scientist's sample request.
	s_c_sampling_code	Code used to identify samples taken for a sample request. Used with s_c_leg.
	sam_archive_working	Part of section where sample was taken. Valid values: WR – whole round, A – archive half, W – working half.
	top_interval	Distance in meters from the top of the section to the top of the sample.
	bottom_interval	Distance in meters from the top of the section to the bottom of the sample.
	piece	Additional identifier for hard rock samples. Each individual piece of rock within a section is numbered consecutively starting at the top of the section.
	sub_piece	Additional identifier for hard rock samples. When a piece is broken, the individual fragments are given consecutive letter designations. Note that subpiece assignments must be made in conjunction with piece numbers.
	beaker_id	The number on the moisture density beaker. Used for samples analyzed for moisture and density.
	volume	Volume of sample.
	entered_by	Indicates who entered the sample into the database.
	sample_depth	Depth of the sample.
	sample_comment	Comment about the sample.
	sam_repository	Repository where sample was taken. Valid values SHIP (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository), WCR (West Coast Repository) and BCR (Bremen Core Repository).
	sam_sample_code_lab	Code to indicate the shipboard lab that will perform the initial analysis.
	sam_section_id	Unique Oracle-generated sequence number to identify each section. This is a foreign key that links a sample to leg, site, hole, core, and section.
	timestamp	Date and time when sample was entered into database. Samples taken before November 25, 1998 and migrated samples have the timestamp 11/25/1998 12:26 PM
System_Type	system_id	Unique identifier for a system of equipment used to collect data.
	system_comments	Comments associated with a piece of analytical equipment
	system_commissioned	Date when a piece of equipment was deployed to collect scientific data for the ODP.
	system_decommissioned	Date when a piece of analytical equipment was no longer used by the ODP.
	system_model_number	The model number of a piece of equipment used for scientific analysis.
	system_name	The name for a piece of equipment used for analysis.

Appendix II. Description of Data Items from XRF query.

Column Name	Column Description	Format
Leg	Number identifying the cruise. The ODP started numbering the scientific cruises of the <i>JR</i> at Leg 101. A leg was nominally two months duration. During the 18+ years of the ODP, there were 110 cruises on the <i>JR</i> .	Integer 3
Site	Number identifying the site. A site is the location where one or more holes were drilled while the ship was positioned over a single acoustic beacon. The <i>JR</i> visited 656 unique sites during the course of the ODP. Some sites were visited multiple times, including some sites originally visited during the Deep Sea Drilling Program for a total of 673 site visits.	Integer 4
Hole	Letter identifying the hole. Multiple holes could be drilled at a single site by pulling the drill pipe above the seafloor, moving the ship some distance away and drilling another hole. The first hole was designated 'A' and additional holes proceeded alphabetically at a given site. Location information for the cruise was determined by hole latitude and longitude. During ODP, there were 1818 holes drilled or deepened.	Text 1
Core	Cores are numbered serially from the top of the hole downward. Cored intervals are up to 9.7 m long, the maximum length of the core barrel. Recovered material was placed at the top of the cored interval, even when recovery was less than 100%. More than 220 km of core were recovered by the ODP.	Integer 3
Type	All cores are tagged by a letter code that identifies the coring method used.	Text 1
Section	Cores are cut into 1.5 m sections in order to make them easier to handle. Sections are numbered serially, with Section 1 at the top of the core. XRF measurements were made on samples taken from sections. Core Catcher sections identified as "CC".	Integer 2 (Text 2)
Top (cm)	The top interval of a measurement in centimeters measured from the top of a section.	Decimal F5.1
Bottom (cm)	The bottom interval of a measurement in centimeters measured from the top of a section.	Decimal F5.1
Depth (mbsf)	Distance in meters from the seafloor to the measurement location.	Decimal F7.3
Run	Run identifier assigned by shipboard scientists or lab technician to identify a given batch of samples.	Text 5
Replicate	Split of a sample	Text 3
Bead Loss on Ignition	Loss on Ignition. The percentage of weight lost after igniting the XRF bead $[(\text{post_ign_sample_wt}/\text{pre_ign_sample_wt}) - 1] * (-100)$.	Decimal F5.2
Silica – SiO ₂ (wt %)	Analytical result for major oxide Silica in weight percent.	Decimal F15.5
Titanium Oxide – TiO ₂ (wt %)	Analytical result for major Titanium oxide in weight percent.	Decimal F15.5
Aluminum Oxide – Al ₂ O ₃ (wt %)	Analytical result for major Aluminum oxide in weight percent.	Decimal F15.5
Iron Oxide – Fe ₂ O ₃ * (wt %)	Analytical result for major Iron oxide in weight percent.	Decimal F15.5
Manganese Oxide – MnO (wt %)	Analytical result for major Manganese oxide in weight percent.	Decimal F15.5
Magnesium Oxide – MgO (wt %)	Analytical result for major Magnesium oxide in weight percent.	Decimal F15.5
Calcium Oxide – CaO (wt %)	Analytical result for major Calcium oxide in weight percent.	Decimal F15.5
Sodium Oxide – Na ₂ O (wt %)	Analytical result for major Sodium oxide in weight percent.	Decimal F15.5
Potassium Oxide – K ₂ O (wt %)	Analytical result for major Potassium oxide in weight percent.	Decimal F15.5
Phosphorus Pentoxide - P ₂ O ₅ (wt %)	Analytical result for major Phosphorus Pentoxide in weight percent.	Decimal F15.5
Niobium - Nb (ppm)	Analytical result for trace element Niobium in parts per million.	Decimal F15.5
Zirconium - Zr (ppm)	Analytical result for trace element Zirconium in parts per million.	Decimal F15.5
Yttrium - Y (ppm)	Analytical result for trace element Yttrium in parts per million.	Decimal F15.5
Sulfur - S (ppm)	Analytical result for trace element Sulfur in parts per million.	Decimal F15.5
Strontium - Sr (ppm)	Analytical result for trace element Strontium in parts per million.	Decimal F15.5
Rubidium - Rb (ppm)	Analytical result for trace element Rubidium in parts per million.	Decimal F15.5
Scandium - Sc (ppm)	Analytical result for trace element Scandium in parts per million.	Decimal F15.5
Molybdenum - Mo (ppm)	Analytical result for trace element Molybdenum in parts per million.	Decimal F15.5
Beryllium - Be (ppm)	Analytical result for trace element Beryllium in parts per million.	Decimal F15.5
Thorium - Th (ppm)	Analytical result for trace element Thorium in parts per million.	Decimal F15.5
Cobalt - Co (ppm)	Analytical result for trace element Cobalt in parts per million.	Decimal F15.5
Gadolinium - Gd (ppm)	Analytical result for trace element Gadolinium in parts per million.	Decimal F15.5

Column Name	Column Description	Format
Dysprosium - Dy (ppm)	Analytical result for trace element Dysprosium in parts per million.	Decimal F15.5
Erbium - Er (ppm)	Analytical result for trace element Erbium in parts per million.	Decimal F15.5
Ytterbium - Yb (ppm)	Analytical result for trace element Ytterbium in parts per million.	Decimal F15.5
Hafnium - Hf (ppm)	Analytical result for trace element Hafnium in parts per million.	Decimal F15.5
Lead - Pb (ppm)	Analytical result for trace element Lead in parts per million.	Decimal F15.5
Gallium - Ga (ppm)	Analytical result for trace element Gallium in parts per million.	Decimal F15.5
Zinc - Zn (ppm)	Analytical result for trace element Zinc in parts per million.	Decimal F15.5
Copper - Cu (ppm)	Analytical result for trace element Copper in parts per million.	Decimal F15.5
Nickel - Ni (ppm)	Analytical result for trace element Nickel in parts per million.	Decimal F15.5
Chromium - Cr (ppm)	Analytical result for trace element Chromium in parts per million.	Decimal F15.5
Vanadium - V (ppm)	Analytical result for trace element Vanadium in parts per million.	Decimal F15.5
Cerium - Ce (ppm)	Analytical result for trace element Cerium in parts per million.	Decimal F15.5
Barium - Ba (ppm)	Analytical result for trace element Barium in parts per million.	Decimal F15.5
Cesium - Cs (ppm)	Analytical result for trace element Cesium in parts per million.	Decimal F15.5
Lanthanum - La (ppm)	Analytical result for trace element Lanthanum in parts per million.	Decimal F15.5
Neodymium - Nd (ppm)	Analytical result for trace element Neodymium in parts per million.	Decimal F15.5
Samarium - Sm (ppm)	Analytical result for trace element Samarium in parts per million.	Decimal F15.5
Sample Type	Type of rock or sediment, e.g., Basalt, Gabbro, Sediment	Text 40
Comment	Comment about the XRF analysis or additional information about the sample type.	Text 80